

OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

DATE: December 8, 2005

SUBJECT: **Reregistration of Dicamba:** Livestock Storage Stability Study and
Ruminant Feeding Study
PC Code: 029801
DP Barcodes: D320564 and D322842
MRIDs: 44891303 and 46668101

REVIEWER: Christine L. Olinger, Chemist
Reregistration Branch I
Health Effects Division (7509C)

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THROUGH: Toiya Goodlow, Chemist
Reregistration Branch I
Health Effects Division (7509C)

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and

Whang Phang Ph.D., Branch Senior Scientist
Reregistration Branch I
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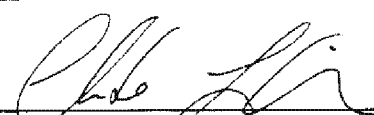
TO: Kendra Tyler
Reregistration Branch I
Health Effects Division (7509C)

BASF Corporation has submitted a livestock commodity storage stability study and a ruminant feeding study in support of the reregistration of the herbicide dicamba. Attached are the data evaluation records for each study. Both studies are adequate and no further livestock storage stability or ruminant feeding studies are required.

cc: COlinger, Reg. Std. File,
7509C:RRB1:CLOlinger:clo:CM#2:Rm 722J:305-5406:11/22/05
RDI: TGoodlow: 11/18/05; WPhang: 11/28/05

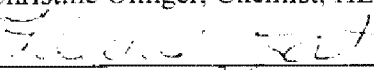
DEC 15 2005

Primary Evaluator


Christine Olinger, Chemist, HED/RRB1

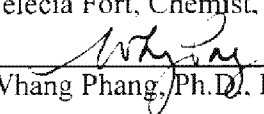
Date: 12/8/05

Peer Reviewer


Felecia Fort, Chemist, HED/RRB1

Date: 12/8/05

Approved by


Whang Phang, Ph.D., BSS, HED/RRB1

Date: 12/08/05

STUDY REPORTS:

MRID No. 44891303; Wofford, J.T., Milinsky, D.S., Riley, M.; (1998) Study title: "A Meat and Milk Magnitude of the Residue Study with Dicamba in Lactating Cows; Lab Project Number: 97076; BASF Registration Document No. 98/5106; Unpublished study prepared by BASF Corporation: 213 pages.

EXECUTIVE SUMMARY:

Dicamba acid was administered via balling gun to five head of cattle for 28 days. Dosing was made at a nominal concentration of 1000 ppm in the feed. Three of the animals were sacrificed within 2 hours of the final dose; two animals were sacrificed five or ten days after the final dose to study the depuration of dicamba in livestock. Samples were analyzed using BASF Analytical Method Number AM-0938-0944-0, with some modifications for fat and cream samples. Acceptable method recoveries were obtained for all matrices. Samples were stored for an interval of demonstrated stability for dicamba and its metabolite 3,6 dichlorosalicylic acid. At sacrifice residues of dicamba and DCSA were detectable in all matrices at levels ranging from 0.4 ppm in muscle to 47 ppm in kidney. Residues in milk reached a plateau within a few days of dosing, with an approximate maximum of 0.2 ppm. Residues in milk dissipated to non-detectable levels within five days of dosing cessation, as observed in the depuration study.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the data depicting residues in livestock are classified as scientifically acceptable.

The acceptability of this study for regulatory purposes will be addressed in the Residue Chemistry Chapter to the Registration Eligibility Decision document.

Signed and dated Good Laboratory Practice (GLP), Quality Assurance and Data Confidentiality statements were provided. Several minor deviations from GLP requirements were noted in the compliance statement, but none of these deviations are likely to impact the validity of the study.



A. BACKGROUND INFORMATION

Dicamba (3,6-dichloro-2-methoxybenzoic acid) is a selective benzoic acid herbicide registered for the control of weeds prior to or before their emergence. Different forms of dicamba (acid and salt) have registered uses on several food/feed crops including asparagus, barley, corn (field and pop), grasses grown in pasture and rangeland, oats, proso millet, rye, sorghum, soybeans, sugarcane, and wheat. Application rates for currently registered products range from 0.5 to 2.8 lb ae/A.

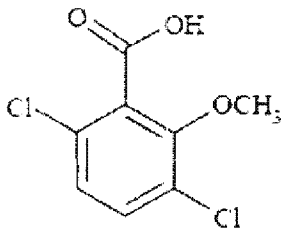
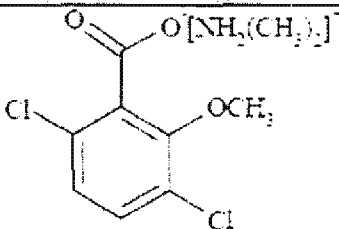
TABLE A.1. Test Compound Nomenclature.	
PC Code 029801	
Chemical structure	
Common name	Dicamba acid
Molecular Formula	$C_8H_6Cl_2O_3$
Molecular Weight	221.04
IUPAC name	3,6-dichloro- <i>o</i> -anisic acid
CAS name	3,6-dichloro-2-methoxybenzoic acid or 2-methoxy-3,6-dichlorobenzoic acid
CAS #	1918-00-9
PC Code 029802	
Chemical structure	
Common name	Dicamba dimethylamine salt (DMA salt)
Molecular Formula	$C_{10}H_{13}Cl_2NO_3$
Molecular Weight	266.1

TABLE A.1. Test Compound Nomenclature.	
CAS #	2300-66-5
PC Code 029806	
Chemical Structure	
Common name	Dicamba sodium salt (Na salt)
Molecular Formula	C ₈ H ₅ Cl ₂ NaO ₃
Molecular Weight	243.0
CAS #	1982-69-0
PC Code 128931	
Chemical Structure	
Common name	Dicamba diglycolamine salt (DGA salt)
Molecular Formula	C ₁₂ H ₁₇ Cl ₂ NO ₅
Molecular Weight	326.18
CAS #	104040-79-1
PC Code 128944	
Chemical Structure	
Common name	Dicamba isopropylamine salt (IPA salt)
Molecular Formula	C ₁₁ H ₁₅ Cl ₂ NO ₃

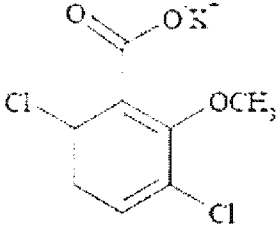
TABLE A.1. Test Compound Nomenclature.	
Molecular Weight	280.15
CAS #	55871-02-8
PC Code 129043	
Chemical Structure	
Common name	Dicamba potassium salt (K salt)
Molecular Formula	C ₈ H ₅ Cl ₂ KO ₃
Molecular Weight	259.1
CAS #	10007-85-9

Table A.2. Physicochemical Properties of Dicamba and its Salts		
Parameter	Value	Reference
Dicamba acid (PC Code 029801)		
Melting point	114-116 °C (PAI) 90-100 °C (87% TGAI)	SRR Reregistration Standard, 6/30/89
pH	2.5-3.0 (87% TGAI)	
Density, bulk density, or specific gravity	1.57 g/mL at 25 °C (87% TGAI)	
Water solubility	0.5 g/100 mL at 25 °C (PAI)	
Solvent solubility	g/100 mL at 25 °C (PAI)	
	dioxane 118.0	
	ethanol 92.2	
	isopropyl alcohol 76.0	
	methylene chloride 26.0	RD D266167, 6/26/00, B. Kitchens
	acetone 17.0	
	toluene 13.0	
	xylene 7.8	
	heavy aromatic naphthalene 5.2	
Vapor pressure	3.4 x 10 ⁻⁵ mm Hg at 25 °C (PAI)	
Dissociation constant, pK _a	1.97 (PAI)	
Octanol/water partition coefficient	0.1 (PAI)	
UV/visible absorption spectrum	neutral: 511 (275 nm) acidic (pH 0-1): 1053 (281 nm) basic (pH 13-14): 469 (274 nm)	



Table A.2. Physicochemical Properties of Dicamba and its Salts		
Parameter	Value	Reference
Dicamba DMA salt (PC Code 029802)		
Melting point	101.0-114.5 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
pH	3.89 at 25 °C (1% solution)	
Density, bulk density, or specific gravity	0.77 g/mL at 25 °C (tap density)	
Water solubility	94.5 g/100 mL at 25 °C	
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	D198000, 5/5/94, P. Deschamp
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	K _{OW} = 0.078	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
UV/visible absorption spectrum	Not available	
Dicamba Na salt (PC Code 029806)		
Melting point	320-325 °C	RD Memorandum, 9/26/94, T. Alston
pH	7.16	
Density, bulk density, or specific gravity	1.03 g/mL at 25 °C	
Water solubility	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the Na salt	D198000, 5/5/94, P. Deschamp
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the Na salt	
UV/visible absorption spectrum	Not available	
Dicamba DGA salt (PC Code 128931)		



Table A.2. Physicochemical Properties of Dicamba and its Salts		
Parameter	Value	Reference
Melting point	52.0-85.0 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
pH	7.60 at 25 °C (1% solution)	
Density, bulk density, or specific gravity	0.69 g/mL at 25 °C (tap density)	
Water solubility	107 g/100 mL at 25 °C	
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	D198000, 5/5/94, P. Deschamp
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	K _{OW} = 0.061	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
UV/visible absorption spectrum	Not available	
Dicamba IPA salt (PC Code 128944)		
Melting point	93.5-127.5 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
pH	4.68 at 25 °C (1% solution)	
Density, bulk density, or specific gravity	0.63 g/mL at 25 °C (tap density)	
Water solubility	59.6 g/100 mL at 25 °C	
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	D198000, 5/5/94, P. Deschamp
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	K _{OW} = 0.070	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
UV/visible absorption spectrum	Not available	
Dicamba K salt (PC Code 129043)		

Table A.2. Physicochemical Properties of Dicamba and its Salts		
Parameter	Value	Reference
Melting point	Decomposes at 213.5 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786. 11/21/95, L. Cheng
pH	8.12 at 25 °C (1% solution)	
Density, bulk density, or specific gravity	0.88 g/mL at 25 °C (tap density)	
Water solubility	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the K salt	
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	D198000, 5/5/94, P. Deschamp
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the K salt	
UV/visible absorption spectrum	Not available	

B. EXPERIMENTAL DESIGN

B.1. Livestock

TABLE B.1.1. Description of Livestock Used in the Feeding Study. ¹					
Animal No.	Breed	Group	Weight at study initiation (kg)	Health status	Description of housing/holding area
1	Holstein	Control	661.024	Acceptable	Outdoor individual sand-floor pens
2	Holstein	Control	616.986	Acceptable	
3	Holstein	Control	622.434	Acceptable	
4	Holstein	1000 ppm	653.306	Acceptable	
5	Holstein	1000 ppm	528.456	Acceptable	
6	Holstein	1000 ppm	694.62	Acceptable	
7	Holstein	1000 ppm	638.324	Acceptable	
8	Holstein	1000 ppm	655.122	Acceptable	

¹ Ages of individual animals were not specified, but were described as ages 3-5 years.

TABLE B.1.2. Test Animal Dietary Regime.				
Animal Number	Average Feed consumption (kg/day)	Water	Acclimation period	Composition of the Diet
1	56.25	<i>Ad libitum</i>	14 days	Alfalfa Hay 24.56% Corn Silage 23.74% Rolled Corn 15.0% Corn Hominy 8.18%
2	46.5	<i>Ad libitum</i>	14 days	
3	52.25	<i>Ad libitum</i>	14 days	
4	46.5	<i>Ad libitum</i>	14 days	



5	48.5	<i>Ad libitum</i>	14 days	Cotton Seed 5.46%
6	54.25	<i>Ad libitum</i>	14 days	Cotton Hulls 1.36%
7	54	<i>Ad libitum</i>	14 days	Soybean Hulls 5.46%
8	51	<i>Ad libitum</i>	14 days	Cotton Seed Meal 5.46% Sodium Bicarbonate 0.15% Mineral 1.64%

TABLE B.1.3. Dosing Regime.

Animal No.	Treatment Type	Average administered dose (mg/day)	Residue intake in diet (ppm)	Vehicle	Timing/ Duration
1	Control	0	0	Cellulose Capsule	30 days
2	Control	0	0	Cellulose Capsule	30 days
3	Control	0	0	Cellulose Capsule	30 days
4	1000 ppm nominal	18.2	911	Cellulose Capsule	30 days
5	1000 ppm nominal	18.7	968	Cellulose Capsule	30 days
6	1000 ppm nominal	21.1	991	Cellulose Capsule	30 days
7	1000 ppm nominal	20.7	963	Cellulose Capsule	30 days
8	1000 ppm nominal	19.8	934	Cellulose Capsule	30 days

TABLE B.1.4 Sample Collection.

Animal No.	Milk Collection Times	Average Daily Milk Production, kg	Interval from last dose to sacrifice, hours	Tissues Harvested and Analyzed
1	AM and PM	20.44	1.13	Muscle, Fat, Kidney, and Liver
2	AM and PM	21.57	1.42	
3	AM and PM	14.90	0.78	
4	AM and PM	16.25	1.75	
5	AM and PM	15.94	1.93	
6	AM and PM	18.40	1.47	
7	AM and PM	23.65	120.43	
8	AM and PM	17.27	264.7	

B.2. Sampling Handling and Preparation

Samples were stored frozen immediately upon collection. Thigh and loin muscle samples were composited after sampling. Other composited samples collected were distal portions of each lobe of the liver; both kidneys; and mesenteric and peripheral fat samples. The tissue samples were sliced, homogenized, and then stored frozen. Tissue samples were thawed prior to analysis. Milk samples were thawed, shaken, and an aliquot was removed for analysis.

B.3. Analytical Methodology

Samples were analyzed using BASF Analytical Method Number AM-0938-0944-0, which can be used to determine both dicamba and 3,6-dichlorosalicylic acid (DCSA) in livestock tissues and milk. Both dicamba and DCSA are converted to the methyl ester of dicamba. Modifications to the method are required for some matrices.

In brief, samples were extracted with 1N HCl, which hydrolyzed the dicamba and DCSA residues. An aliquot was removed for further sample preparation. The residues were partitioned into ethyl acetate and then methylated using diazomethane. The extract was cleaned up on a silica gel SPE column. Determination of the methylated residues was by gas chromatography with an electron capture detector. Fat and cream samples required an additional clean-up step. After hydrolysis, 4N KOH was added to the extract, and the mixture was allowed to precipitate overnight. An aliquot of the extract was then subjected to esterification clean-up, followed by analysis as previously described. The limit of quantitation of dicamba and DCSA was 0.01 ppm. Recovery samples were analyzed alongside the samples from the feeding study.

C. RESULTS AND DISCUSSION

Recovery samples were analyzed alongside the samples from the feeding study. Fortification levels generally bracketed the residue levels found in the study samples. The results of all fortified samples are found in Table C.1. The registrant corrected the residues determined in the fortification samples by subtracting the residues found in the control, but in general, residues were not found in control samples. Recoveries varied considerably in some matrices, but were generally within acceptable levels. Treated samples were not corrected for percent recovery.

A summary of the sample storage conditions may be found in Table C.2. The maximum storage interval for any commodity was 253 days. A storage stability study (MRID 46668101, DP Barcode D322842, C. Olinger) has demonstrated stability of dicamba and DCSA in ruminant matrices for at least 18 months.

Results of the feeding study may be found in Table C.3 and C.4, and a graph of the concentration of dicamba residues in milk is presented in Figure 1. Only one dose level was tested, nominal 1000 ppm, in response to an Agency request. The amount of pesticide dosed varied from week to week, because the amount of dicamba placed in the capsule was based on the previous week's feed consumption for each animal.

Residue levels in the fat of the animals sacrificed the day of the final dose ranged from 0.202 ppm to 0.511 ppm; for kidney, 9.81 to 46.6 ppm; for liver 2.43 to 5.06 ppm, and for muscle 0.107 to 0.392 ppm. One kidney and one liver sample were analyzed two or three times, and considerable variability was found, which was consistent with the variable recoveries found.

The registrant conducted a depuration study on two animals, sacrificing one five days after the final dose, and the second one ten days after the final dose. Residue levels in milk were below the limit of quantitation within three days of the final dose. At sacrifice the residue levels in both animals had reduced considerably, and were non-detectable in muscle.

TABLE C.1. Summary of Concurrent Recoveries of Dicamba and DCSA from Ruminant Commodities				
Matrix	Spike level (ppm)	Sample size (n)	Recoveries (%)	Mean \pm std dev (%)
Whole Milk – Dicamba	0.01 ppm	8	87, 83, 65, 105, 98, 95, 91, 113	92.1 \pm 14.6

**TABLE C.1. Summary of Concurrent Recoveries of Dicamba and DCSA from Ruminant Commodities**

Matrix	Spike level (ppm)	Sample size (n)	Recoveries (%)	Mean \pm std dev (%)
Whole Milk – Dicamba	1.0 ppm	8	99, 89, 68, 93, 97, 95, 89, 112	92.8 \pm 12.4
Whole Milk – DCSA	0.01 ppm	6	79, 71, 83, 84, 97, 119	88.8 \pm 17.0
Whole Milk – DCSA	1.0 ppm	6	77, 86, 69, 89, 83, 73	79.5 \pm 7.8
Skim Milk – Dicamba	0.01 ppm	1	113	N/A
Skim Milk – Dicamba	1.0 ppm	1	103	N/A
Skim Milk – DCSA	0.01 ppm	1	78	N/A
Skim Milk – DCSA	1.0 ppm	1	71	N/A
Cream – Dicamba	0.01 ppm	1	72	N/A
Cream – Dicamba	1.0 ppm	1	127	N/A
Cream – DCSA	0.01 ppm	1	80	N/A
Cream – DCSA	1.0 ppm	1	128	N/A
Fat – Dicamba	0.01 ppm	1	78	N/A
Fat – Dicamba	1.0 ppm	1	123	N/A
Fat – DCSA	0.01 ppm	1	61	N/A
Fat – DCSA	1.0 ppm	1	120	N/A
Kidney – Dicamba	0.01 ppm	1	66	N/A
Kidney – Dicamba	50.0 ppm	1	78	N/A
Kidney – DCSA	0.01 ppm	1	60	N/A
Kidney – DCSA	1.0 ppm	1	76	N/A
Liver – Dicamba	0.01 ppm	2	116, 101	108 \pm 10.6
Liver – Dicamba	1.0 ppm	1	117	N/A
Liver – Dicamba	10.0 ppm	1	65	N/A
Liver – DCSA	0.01 ppm	1	94	N/A
Liver – DCSA	5.0 ppm	1	84	N/A
Muscle – Dicamba	0.01 ppm	1	56	N/A
Muscle – Dicamba	1.0 ppm	1	63	N/A
Muscle – DCSA	0.01 ppm	1	70	N/A
Muscle – DCSA	5.0 ppm	1	62	N/A

TABLE C.2. Summary of Storage Conditions.

Matrix	Storage Temperature (°C)	Maximum Storage Duration (Days)	Interval of Demonstrated Storage Stability (Days)
Whole Milk	≤ -10 C	190	540
Skim Milk	≤ -10 C	232	540
Cream	≤ -10 C	203	540
Fat	≤ -10 C	189	540
Kidney	≤ -10 C	253	540
Liver	≤ -10 C	233	540
Muscle	≤ -10 C	220	540

TABLE C.3. Residue Data from Cattle Feeding Study with Dicamba at 1000 ppm (Nominal)

Animal identification No.	Matrix/Collection Day	Average Feeding Level (ppm)	Combined Residues of Dicamba and DCSA (ppm)
4	Whole Milk – Day 1	911	0.071
5	Whole Milk – Day 1	968	0.137
6	Whole Milk – Day 1	991	0.073
7	Whole Milk – Day 1	963	0.079
8	Whole Milk – Day 1	934	0.152
8	Whole Milk – Day 1	934	0.224
4	Whole Milk – Day 2	911	0.115
5	Whole Milk – Day 2	968	0.173



TABLE C.3. Residue Data from Cattle Feeding Study with Dicamba at 1000 ppm (Nominal)			
Animal identification No.	Matrix/Collection Day	Average Feeding Level (ppm)	Combined Residues of Dicamba and DCSA (ppm)
6	Whole Milk – Day 2	991	0.095
7	Whole Milk – Day 2	963	0.068
8	Whole Milk – Day 2	934	0.124
4	Whole Milk – Day 4	911	0.111
5	Whole Milk – Day 4	968	0.151
6	Whole Milk – Day 4	991	0.054
7	Whole Milk – Day 4	963	0.046
8	Whole Milk – Day 4	934	0.052
8	Whole Milk – Day 4	934	0.185
4	Whole Milk – Day 7	911	0.054
5	Whole Milk – Day 7	968	0.111
6	Whole Milk – Day 7	991	0.077
7	Whole Milk – Day 7	963	0.073
8	Whole Milk – Day 7	934	0.095
4	Whole Milk – Day 10	911	0.144
5	Whole Milk – Day 10	968	0.084
6	Whole Milk – Day 10	991	0.071
7	Whole Milk – Day 10	963	0.092
8	Whole Milk – Day 10	934	0.085
4	Whole Milk – Day 14	911	0.084
4	Whole Milk – Day 14	911	0.225
5	Whole Milk – Day 14	968	0.019
5	Whole Milk – Day 14	968	0.163
6	Whole Milk – Day 14	991	0.079
7	Whole Milk – Day 14	963	0.081
8	Whole Milk – Day 14	934	0.093
4	Whole Milk – Day 17	911	0.261
5	Whole Milk – Day 17	968	0.155
6	Whole Milk – Day 17	991	0.101
7	Whole Milk – Day 17	963	0.111
8	Whole Milk – Day 17	934	0.097
4	Whole Milk – Day 21	911	0.386
4	Whole Milk – Day 21	911	0.461
4	Whole Milk – Day 21	911	0.506
5	Whole Milk – Day 21	968	0.177
6	Whole Milk – Day 21	991	0.134
7	Whole Milk – Day 21	963	0.151
8	Whole Milk – Day 21	934	0.134
4	Whole Milk – Day 24	911	0.297
5	Whole Milk – Day 24	968	0.116
6	Whole Milk – Day 24	991	0.128
7	Whole Milk – Day 24	963	0.076
8	Whole Milk – Day 24	934	0.213
4	Whole Milk – Day 28	911	0.241
5	Whole Milk – Day 28	968	0.186
6	Whole Milk – Day 28	991	0.151
7	Whole Milk – Day 28	963	<0.01
7	Whole Milk – Day 28	963	0.107
8	Whole Milk – Day 28	934	0.126
7	Whole Milk – Day 32	963	0.051
8	Whole Milk – Day 32	934	0.016
7	Whole Milk – Day 33	963	<0.01



TABLE C.3. Residue Data from Cattle Feeding Study with Dicamba at 1000 ppm (Nominal)			
Animal identification No	Matrix/Collection Day	Average Feeding Level (ppm)	Combined Residues of Dicamba and DCSA (ppm)
8	Whole Milk – Day 33	934	<0.01
7	Whole Milk – Day 35	963	<0.01
8	Whole Milk – Day 35	934	<0.01
8	Whole Milk – Day 37	934	<0.01
8	Whole Milk – Day 39	934	<0.01
4	Skim Milk – Day 30	911	0.191
5	Skim Milk – Day 30	968	0.139
6	Skim Milk – Day 30	991	0.167
7	Skim Milk – Day 30	963	0.138
8	Skim Milk – Day 30	934	0.165
4	Cream – Day 30	911	0.164
5	Cream – Day 30	968	0.141
6	Cream – Day 30	991	0.151
7	Cream – Day 30	963	0.165
8	Cream – Day 30	934	0.150
4	Fat – Day 30	911	0.238
5	Fat – Day 30	968	0.202
6	Fat – Day 30	991	0.511
7	Fat – Day 35	963	<0.01
8	Fat – Day 40	934	0.016
4	Kidney – Day 30	911	9.81
5	Kidney – Day 30	968	11.64
6	Kidney – Day 30	991	46.64
6	Kidney – Day 30	991	37.88
7	Kidney – Day 35	963	0.041
8	Kidney – Day 40	934	0.026
4	Liver – Day 30	911	3.40
5	Liver – Day 30	968	3.26
6	Liver – Day 30	991	5.06
6	Liver – Day 30	911	2.66
6	Liver – Day 30	911	2.43
7	Liver – Day 35	963	0.013
8	Liver – Day 40	934	0.22
4	Muscle – Day 30	911	0.136
5	Muscle – Day 30	968	0.107
6	Muscle – Day 30	991	0.392
7	Muscle – Day 35	963	<0.01
8	Muscle – Day 40	934	<0.01

TABLE C.4. Summary of Residue Data from Ruminant Feeding Study with Dicamba ¹							
Matrix	Feeding Level (ppm)	Residue Levels (ppm)					
		n	Min.	Max.	Median (STMdR)	Mean (STMR)	Std. Dev.
Fat	1000	3	0.202	0.511	0.238	0.317	0.1697
Kidney	1000	3	9.81	46.6	11.6	21.2	18.2
Liver	1000	3	2.43	5.06	3.38	3.35	0.0765
Muscle	1000	3	0.107	0.392	0.136	0.212	0.157

Note that only the samples from cattle sacrificed on the final day of dosing were included in these calculations.

¹If a sample was analyzed more than once, then an average of the analyses for the individual sample was used in calculating the median, mean, and standard deviation.



D. CONCLUSION

An appropriate analytical method was used in this cattle feeding study, conducted at a single feeding level, nominal 1000 ppm. Samples were stored for an interval of demonstrated stability. Residues were generally detectable in all matrices tested. The depuration study conducted in association with this feeding study showed that residues dissipated to non-detectable levels in milk within five days.

E. REFERENCES

The following memorandum was referenced in this review.

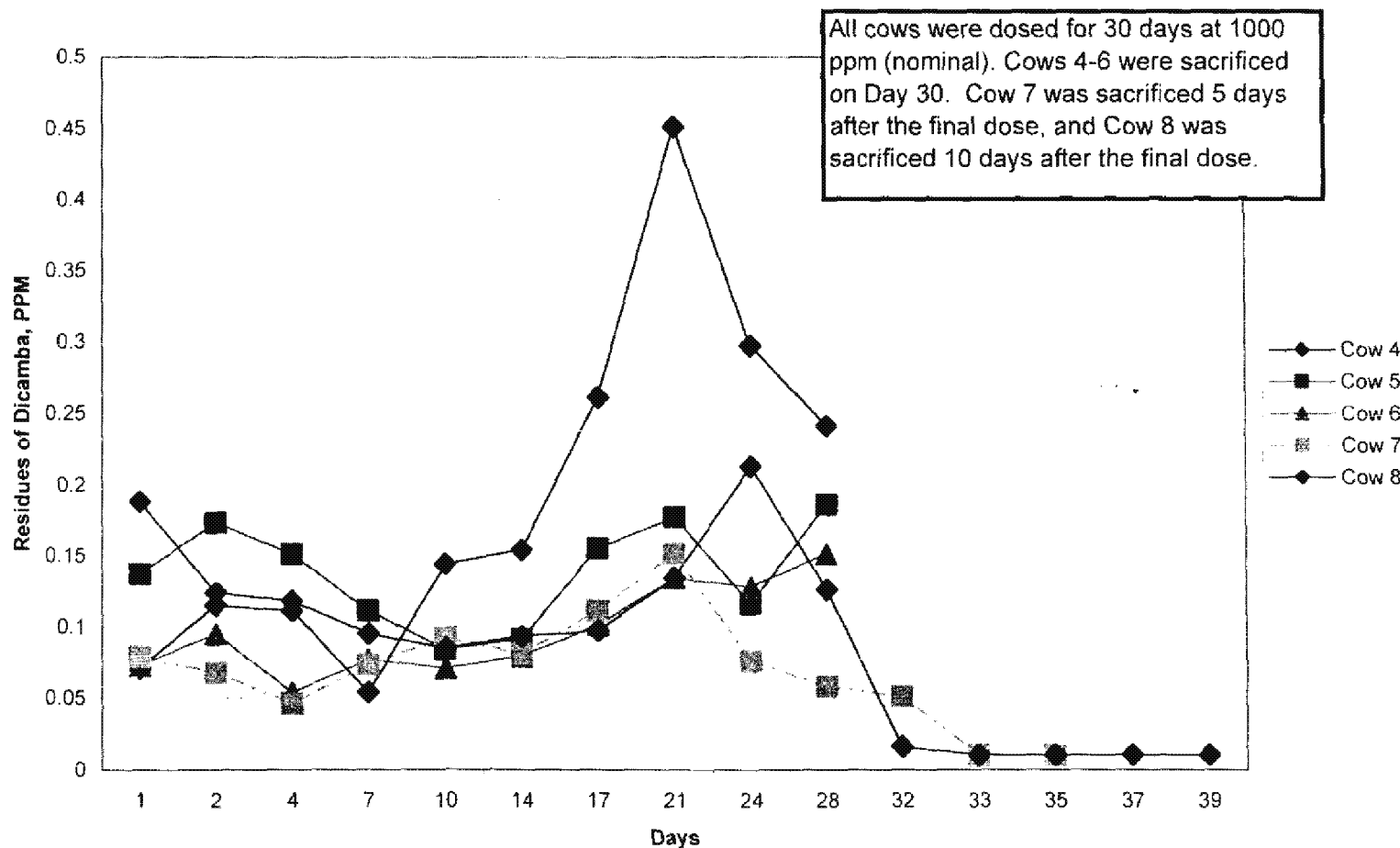
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From: C. Olinger
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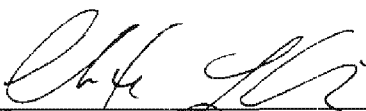
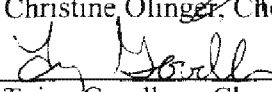
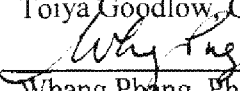
F. DOCUMENT TRACKING

RDI: COlinger (11/22/05); FFort (11/18/05); WPhang (11/28/05); etc.
DP Barcode(s): D320564
PC Code: 029801

Template Version June 2005.

Figure C1. Dicamba Residues in Whole Milk from Cows Dosed with Dicamba at 1000 PPM



Primary Evaluator	 Christine Olinger, Chemist, HED/RRB1	Date: 12/8/05
Peer Reviewer	 Toiya Goodlow, Chemist, HED/RRB1	Date: 12/8/05
Approved by	 Whang Phang, Ph.D., HED/RRB1	Date: 12/08/05

STUDY REPORTS:

MRID No. 46668101; Formanski, L. J. (1996) "Stability of Dicamba and 3,6-Dichlorosalicylic Acid in Stored Frozen Beef Tissues and Milk". Lab Project Number: 480068. Report No. 151. Study No. DP-304489. Unpublished study prepared by Sandoz Agro, Inc. 275 pages.

EXECUTIVE SUMMARY:

Samples of cattle liver, kidney, muscle, fat, and milk spiked with dicamba and the metabolite 3,6-dichlorosalicylic acid (DCSA) at a level of 0.1 ppm each were stored at <-10°C for a duration of 18 months. Samples were analyzed using BASF Analytical Method Number AM-0938-0944-0, with some modifications. Acceptable method recoveries were obtained for all matrices. Residues of parent and metabolite appeared to be stable in all matrices tested for at least 18 months when stored frozen at <-10°C.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the storage stability data are classified as scientifically acceptable.

The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary.

COMPLIANCE:

Signed and dated Good Laboratory Practice (GLP), Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported.



A. BACKGROUND INFORMATION

Dicamba (3,6-dichloro-2-methoxybenzoic acid) is a selective benzoic acid herbicide registered for the control of weeds prior to or before their emergence. Different forms of dicamba (acid and salt) have registered uses on several food/feed crops including asparagus, barley, corn (field and pop), grasses grown in pasture and rangeland, oats, proso millet, rye, sorghum, soybeans, sugarcane, and wheat. Application rates for products currently registered range from 0.5 to 2.8 lb ae/A.

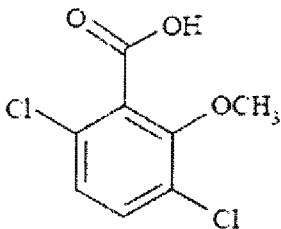
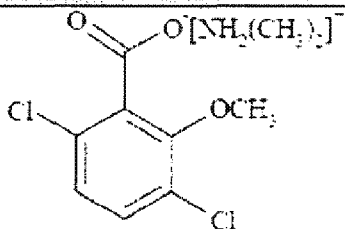
TABLE A.1. Test Compound Nomenclature.	
PC Code 029801	
Chemical structure	
Common name	Dicamba acid
Molecular Formula	C ₈ H ₆ Cl ₂ O ₃
Molecular Weight	221.04
IUPAC name	3,6-dichloro- <i>o</i> -anisic acid
CAS name	3,6-dichloro-2-methoxybenzoic acid or 2-methoxy-3,6-dichlorobenzoic acid
CAS #	1918-00-9
PC Code 029802	
Chemical structure	
Common name	Dicamba dimethylamine salt (DMA salt)
Molecular Formula	C ₁₀ H ₁₃ Cl ₂ NO ₃
Molecular Weight	266.1
CAS #	2300-66-5

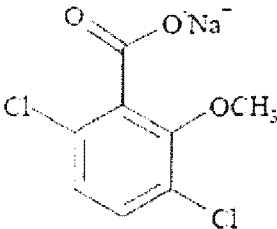
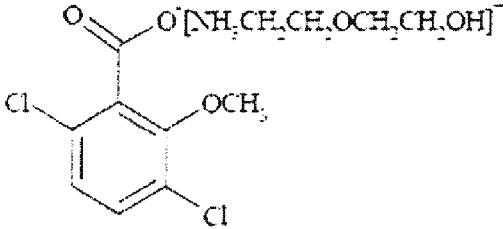
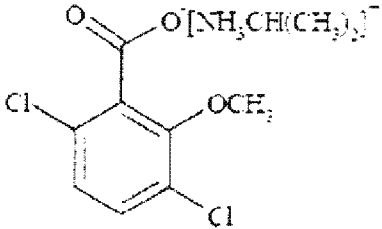
TABLE A.1. Test Compound Nomenclature.	
PC Code 029806	
Chemical Structure	
Common name	Dicamba sodium salt (Na salt)
Molecular Formula	C ₈ H ₅ Cl ₂ NaO ₃
Molecular Weight	243.0
CAS #	1982-69-0
PC Code 128931	
Chemical Structure	
Common name	Dicamba diglycolamine salt (DGA salt)
Molecular Formula	C ₁₂ H ₁₇ Cl ₂ NO ₅
Molecular Weight	326.18
CAS #	104040-79-1
PC Code 128944	
Chemical Structure	
Common name	Dicamba isopropylamine salt (IPA salt)
Molecular Formula	C ₁₁ H ₁₅ Cl ₂ NO ₃
Molecular Weight	280.15

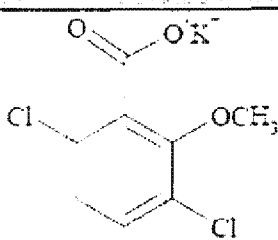
TABLE A.1. Test Compound Nomenclature.	
CAS #	55871-02-8
PC Code 129043	
Chemical Structure	
Common name	Dicamba potassium salt (K salt)
Molecular Formula	C ₈ H ₅ Cl ₂ KO ₃
Molecular Weight	259.1
CAS #	10007-85-9

Table A.2. Physicochemical Properties of Dicamba and its Salts			
Parameter	Value	Reference	
Dicamba acid (PC Code 029801)			
Melting point	114-116 °C (PAI) 90-100 °C (87% TGA1)	SRR Reregistration Standard, 6/30/89	
pH	2.5-3.0 (87% TGA1)		
Density, bulk density, or specific gravity	1.57 g/mL at 25 °C (87% TGA1)		
Water solubility	0.5 g/100 mL at 25 °C (PAI)		
Solvent solubility	g/100 mL at 25 °C (PAI)		
	dioxane		118.0
	ethanol		92.2
	isopropyl alcohol		76.0
	methylene chloride		26.0
	acetone		17.0
	toluene	13.0	
	xylene	7.8	
heavy aromatic naphthalene	5.2		
Vapor pressure	3.4 x 10 ⁻³ mm Hg at 25 °C (PAI)	RD D266167, 6/26/00, B. Kitchens	
Dissociation constant, pK _a	1.97 (PAI)		
Octanol/water partition coefficient	0.1 (PAI)		
UV/visible absorption spectrum	neutral: 511 (275 nm) acidic (pH 0-1): 1053 (281 nm) basic (pH 13-14): 469 (274 nm)		
Dicamba DMA salt (PC Code 029802)			

Table A.2. Physicochemical Properties of Dicamba and its Salts		
Parameter	Value	Reference
Melting point	101.0-114.5 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
pH	3.89 at 25 °C (1% solution)	
Density, bulk density, or specific gravity	0.77 g/mL at 25 °C (tap density)	
Water solubility	94.5 g/100 mL at 25 °C	
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	D198000, 5/5/94, P. Deschamp
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	K _{ow} = 0.078	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
UV/visible absorption spectrum	Not available	
Dicamba Na salt (PC Code 029806)		
Melting point	320-325 °C	RD Memorandum, 9/26/94, T. Alston
pH	7.16	
Density, bulk density, or specific gravity	1.03 g/mL at 25 °C	
Water solubility	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the Na salt	D198000, 5/5/94, P. Deschamp
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the Na salt	
UV/visible absorption spectrum	Not available	
Dicamba DGA salt (PC Code 128931)		
Melting point	52.0-85.0 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng

Table A.2. Physicochemical Properties of Dicamba and its Salts		
Parameter	Value	Reference
pH	7.60 at 25 °C (1% solution)	
Density, bulk density, or specific gravity	0.69 g/mL at 25 °C (tap density)	
Water solubility	107 g/100 mL at 25 °C	
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	D198000, 5/5/94, P. Deschamp
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	K _{OW} = 0.061	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
UV/visible absorption spectrum	Not available	
Dicamba IPA salt (PC Code 128944)		
Melting point	93.5-127.5 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
pH	4.68 at 25 °C (1% solution)	
Density, bulk density, or specific gravity	0.63 g/mL at 25 °C (tap density)	
Water solubility	59.6 g/100 mL at 25 °C	
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	D198000, 5/5/94, P. Deschamp
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	K _{OW} = 0.070	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
UV/visible absorption spectrum	Not available	
Dicamba K salt (PC Code 129043)		
Melting point	Decomposes at 213.5 °C	D213276, D216855, D216859, D216853, D216857, D216862, D217061, D218789, D218792, D218784, D218787, and D218786, 11/21/95, L. Cheng
pH	8.12 at 25 °C (1% solution)	



Table A.2. Physicochemical Properties of Dicamba and its Salts		
Parameter	Value	Reference
Density, bulk density, or specific gravity	0.88 g/mL at 25 °C (tap density)	
Water solubility	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the K salt	D198000, 5/5/94, P. Deschamp
Solvent solubility	N/A; data for the free acid are representative of the dicamba salts	
Vapor pressure		
Dissociation constant, pK _a		
Octanol/water partition coefficient	N/A; data for the organic salts (DMA, DGA, and IPA) are representative of the K salt	
UV/visible absorption spectrum	Not available	

B. EXPERIMENTAL DESIGN

B.1. Sample Handling and Preparation

Ten gram subsamples of beef fat, muscle, kidney, liver, and milk were weighed into separate glass bottles and fortified with both dicamba and 3,6-dichlorosalicylic acid (DCSA) at a concentration of 0.1 ppm each. The fortified subsamples were stored frozen at <10°C. Control samples were stored as well. Three subsamples were removed at 1 day, 3 months, 6 months, 12 months, and 18 months. Two control samples were removed at each interval as well and freshly fortified for determining the method recovery.

B.2. Analytical Methodology

Samples were analyzed using BASF Analytical Method Number AM-0938-0944-0, which can be used to determine both dicamba and 3,6-dichlorosalicylic acid (DCSA) in livestock tissues and milk. Some modifications to the method were made, including derivitization with diazobutane twice.

In brief, samples are extracted with 1N HCl, which hydrolyzes the dicamba and DCSA residues. An aliquot is removed for further sample preparation. The residues are partitioned into ethyl ether and then butylated using diazobutane. The extract was cleaned up on a silica gel column. Determination of the methylated residues was performed by gas chromatography with an electron capture detector. The extracts were also analyzed using gas chromatography with mass selective detection. The limit of quantitation of dicamba and DCSA was 0.01 ppm



A. RESULTS AND DISCUSSION

A summary of the concurrent recovery samples from each storage interval is presented in Table C.1. In general, higher recoveries were obtained using the GC/MSD analysis than the GC/ECD analysis. Although the results for some matrices show high variability, the average method recoveries are within acceptable limits.

Results of the storage stability studies for dicamba and DCSA are presented in Table C.2. Average recoveries at some intervals for a few matrices are relatively low, but are acceptable when corrected for the concurrent recovery values.

TABLE C.1. Summary of Concurrent Recoveries of Dicamba and DCSA from Ruminant Commodities				
Matrix	Spike level (ppm)	Sample size (n)	Recoveries (%)	Mean ± std dev (%)
Dicamba				
Beef Kidney – GC/ECD	0.1	10	99, 104, 62, 69, 74, 75, 53, 61, 57, 79	73.3 ± 17.0
Beef Kidney – GC/MSD	0.1	10	101, 102, 72, 83, 80, 84, 105, 96, 114, 105	94.2 ± 13.6
Beef Liver – GC/ECD	0.1	10	100, 83, 63, 60, 68, 75, 67, 65, 70, 72	72.3 ± 11.7
Beef Liver – GC/MSD	0.1	10	89, 81, 73, 75, 72, 77, 116, 111, 99, 99	89.2 ± 16.2
Milk – GC/ECD	0.1	10	104, 100, 92, 89, 73, 69, 86, 87, 75, 84	85.9 ± 11.3
Milk – GC/MSD	0.1	10	105, 115, 84, 86, 85, 73, 98, 100, 91, 94	93.1 ± 12.0
Beef Muscle – GC/ECD	0.1	10	113, 114, 75, 75, 73, 77, 80, 74, 75, 75	83.1 ± 16.1
Beef Muscle – GC/MSD	0.1	10	108, 105, 84, 87, 93, 87, 93, 93, 98, 104	95.2 ± 8.3
Beef Fat – GC/ECD	0.1	10	97, 93, 81, 94, 79, 87, 85, 94, 87, 83	88 ± 6.2
Beef Fat – GC/MSD	0.1	10	119, 112, 84, 93, 107, 113, 89, 123, 126, 103	107 ± 14.5
DCSA				
Beef Kidney – GC/ECD	0.1	10	110, 105, 58, 75, 66, 67, 60, 48, 56, 64	70.9 ± 20.6
Beef Kidney – GC/MSD	0.1	10	78, 83, 64, 70, 76, 84, 91, 115, 100, 101	86.2 ± 15.7
Beef Liver – GC/ECD	0.1	10	116, 106, 65, 65, 68, 63, 74, 73, 75, 68	77.3 ± 18.4
Beef Liver – GC/MSD	0.1	10	70, 62, 61, 64, 65, 66, 119, 108, 92, 97	80.4 ± 21.6
Milk – GC/ECD	0.1	10	89, 92, 71, 79, 75, 65, 79, 77, 75, 84	78.6 ± 8.1
Milk – GC/MSD	0.1	10	82, 89, 75, 77, 84, 70, 94, 96, 75, 87	82.9 ± 8.7
Beef Muscle – GC/ECD	0.1	10	100, 106, 59, 59, 65, 66, 51, 56, 78, 66	70.6 ± 18.6
Beef Muscle – GC/MSD	0.1	10	90, 82, 76, 77, 93, 91, 84, 93, 108, 94	88.8 ± 9.5



TABLE C.1. Summary of Concurrent Recoveries of Dicamba and DCSA from Ruminant Commodities				
Matrix	Spike level (ppm)	Sample size (n)	Recoveries (%)	Mean \pm std dev (%)
Beef Fat – GC/ECD	0.1	10	90, 91, 54, 59, 74, 77, 73, 70, 99, 91	77.8 \pm 14.8
Beef Fat – GC/MSD	0.1	10	97, 102, 73, 80, 91, 108, 97, 122, 135, 115	102 \pm 18.8

TABLE C.2. Stability of Dicamba and DCSA Residues in Ruminant Commodities Following Storage at -10°C.						
Commodity	Spike level (ppm)	Storage interval Months	Recovered residues (ppm)	Mean Recovered Residues (ppm)	Mean Recovery (%)	Corrected % recovery*
Dicamba						
Beef Kidney - GC/ECD	0.1	1 day	0.098, 0.096, 0.092	0.0953	95.3	95.3
	0.1	3	0.074, 0.074	0.074	74	112
	0.1	6	0.073, 0.083, 0.075	0.077	77	104
	0.1	12	0.053, 0.061, 0.054	0.056	56	98.2
	0.1	18	0.074, 0.074, 0.079	0.0757	75.7	95.8
Beef Kidney – GC/MSD	0.1	1 day	0.106, 0.115, 0.094	0.11	105	105
	0.1	3	0.081, 0.083	0.08	82	105
	0.1	6	0.078, 0.088, 0.078	0.08	81.3	99.2
	0.1	12	0.106, 0.098, 0.091	0.10	98.3	98.3
	0.1	18	0.117, 0.121, 0.11	0.12	116	116
Beef Liver– GC/ECD	0.1	1 day	0.094, 0.098, 0.095	0.0957	95.7	99.7
	0.1	3	0.071, 0.069, 0.068	0.0693	69.3	112
	0.1	6	0.068, 0.07, 0.075	0.0710	71.0	98.6
	0.1	12	0.07, 0.075, 0.071	0.0720	72.0	109
	0.1	18	0.067, 0.071, 0.068	0.0687	68.7	96.7
Beef Liver– GC/MSD	0.1	1 day	0.094, 0.094, 0.094	0.0940	94.0	111
	0.1	3	0.082, 0.08, 0.078	0.0800	80.0	108
	0.1	6	0.081, 0.073, 0.073	0.0757	75.7	102
	0.1	12	0.106, 0.112, 0.11	0.109	109	109
	0.1	18	0.102, 0.104, 0.106	0.104	104	104
Milk– GC/ECD	0.1	1 day	0.112, 0.11, 0.099	0.107	107	107
	0.1	3	0.086, 0.093, 0.091	0.0900	90.0	100
	0.1	6	0.074, 0.071, 0.068	0.0710	71.0	100
	0.1	12	0.086, 0.072, 0.073	0.0770	77.0	89.5
	0.1	18	0.061, 0.086, 0.06	0.0690	69.0	86.3
Milk– GC/MSD	0.1	1 day	0.109, 0.111, 0.093	0.104	104	104
	0.1	3	0.085, 0.088, 0.093	0.0887	88.7	104
	0.1	6	0.081, 0.083, 0.084	0.0827	82.7	105
	0.1	12	0.102, 0.096, 0.11	0.103	103	104
	0.1	18	0.087, 0.097, 0.118	0.101	101	109
Beef Muscle– GC/ECD	0.1	1 day	0.114, 0.113, 0.122	0.116	116	116
	0.1	3	0.084, 0.077, 0.084	0.0817	81.7	109
	0.1	6	0.084, 0.079, 0.078	0.0803	80.3	107
	0.1	12	0.083, 0.086, 0.076	0.0817	81.7	106
	0.1	18	0.059, 0.078, 0.064	0.0670	67.0	89.3
Beef Muscle– GC/MSD	0.1	1 day	0.114, 0.112, 0.119	0.115	115	115
	0.1	3	0.093, 0.09, 0.099	0.0940	94.0	109
	0.1	6	0.095, 0.09, 0.09	0.0917	91.7	102
	0.1	12	0.106, 0.103, 0.107	0.105	105	113
	0.1	18	0.08, 0.101, 0.117	0.0993	99.3	99.3



TABLE C.2. Stability of Dicamba and DCSA Residues in Ruminant Commodities Following Storage at -10°C.						
Commodity	Spike level (ppm)	Storage interval Months	Recovered residues (ppm)	Mean Recovered Residues (ppm)	Mean Recovery (%)	Corrected % recovery*
Beef Fat–GC/ECD	0.1	1 day	0.09, 0.099, 0.092	0.0937	93.7	98.6
	0.1	3	0.09, 0.089, 0.088	0.0890	89.0	102
	0.1	6	0.087, 0.085, 0.074	0.0820	82.0	98.8
	0.1	12	0.082, 0.084, 0.091	0.0857	85.7	95.2
	0.1	18	0.084, 0.092, 0.085	0.0870	87.0	102
Beef Fat–GC/MSD	0.1	1 day	0.124, 0.132, 0.107	0.121	121	121
	0.1	3	0.097, 0.094, 0.089	0.0933	93.3	106
	0.1	6	0.091, 0.089, 0.102	0.0940	94.0	94.0
	0.1	12	0.11, 0.129, 0.123	0.121	121	121
	0.1	18	0.116, 0.112, 0.116	0.115	115	115
DCSA						
Beef Kidney – GC/ECD	0.1	1 day	0.111, 0.112, 0.108	0.110	110.33	110.33
	0.1	3	0.067, 0.068	0.0675	67.50	102.27
	0.1	6	0.062, 0.068, 0.065	0.0650	65.00	98.48
	0.1	12	0.056, 0.055, 0.05	0.0537	53.67	99.38
	0.1	18	0.069, 0.076, 0.074	0.0730	73.00	114.06
Beef Kidney – GC/MSD	0.1	1 day	0.082, 0.088, 0.083	0.0843	84.33	105.42
	0.1	3	0.068, 0.07	0.0690	69.00	102.99
	0.1	6	0.074, 0.083, 0.078	0.0783	78.33	97.92
	0.1	12	0.092, 0.115, 0.099	0.102	102.00	102.00
	0.1	18	0.104, 0.105, 0.103	0.104	104.00	104.00
Beef Liver–GC/ECD	0.1	1 day	0.113, 0.11, 0.103	0.109	109	109
	0.1	3	0.068, 0.064, 0.061	0.0643	64.3	99.0
	0.1	6	0.06, 0.062, 0.066	0.0627	62.7	95.0
	0.1	12	0.07, 0.06, 0.071	0.0670	67.0	90.5
	0.1	18	0.066, 0.074, 0.078	0.0727	72.7	101
Beef Liver–GC/MSD	0.1	1 day	0.074, 0.076, 0.074	0.0747	74.7	113
	0.1	3	0.059, 0.062, 0.064	0.0617	61.7	99.5
	0.1	6	0.07, 0.066, 0.069	0.0683	68.3	104
	0.1	12	0.123, 0.119, 0.108	0.117	117	117
	0.1	18	0.093, 0.093, 0.091	0.0923	92.3	98.2
Milk– GC/ECD	0.1	1 day	0.095, 0.101, 0.091	0.0957	95.7	106
	0.1	3	0.066, 0.072, 0.074	0.0707	70.7	94.2
	0.1	6	0.065, 0.063, 0.065	0.0643	64.3	90.6
	0.1	12	0.091, 0.072, 0.081	0.0813	81.3	104
	0.1	18	0.071, 0.083, 0.071	0.0750	75.0	93.8
Milk– GC/MSD	0.1	1 day	0.087, 0.088, 0.073	0.0827	82.7	96.1
	0.1	3	0.064, 0.077, 0.076	0.0723	72.3	95.2
	0.1	6	0.075, 0.07, 0.073	0.0727	72.7	94.4
	0.1	12	0.096, 0.088, 0.105	0.0963	96.3	101
	0.1	18	0.076, 0.082, 0.091	0.0830	83.0	102
Beef Muscle–GC/ECD	0.1	1 day	0.092, 0.094, 0.091	0.0923	92.3	92.3
	0.1	3	0.061, 0.059, 0.057	0.0590	59.0	100
	0.1	6	0.055, 0.06, 0.062	0.0590	59.0	89.4
	0.1	12	0.057, 0.058, 0.055	0.0567	56.7	105
	0.1	18	0.054, 0.067, 0.074	0.0650	65.0	90.3
Beef Muscle–GC/MSD	0.1	1 day	0.087, 0.093, 0.096	0.0920	92.0	107
	0.1	3	0.074, 0.07, 0.078	0.0740	74.0	97.4
	0.1	6	0.08, 0.08, 0.073	0.0777	77.7	84.4
	0.1	12	0.089, 0.093, 0.093	0.0917	91.7	105



TABLE C.2. Stability of Dicamba and DCSA Residues in Ruminant Commodities Following Storage at -10°C.						
Commodity	Spike level (ppm)	Storage interval Months	Recovered residues (ppm)	Mean Recovered Residues (ppm)	Mean Recovery (%)	Corrected % recovery*
Beef Fat–GC/ECD	0.1	18	0.072, 0.091, 0.093	0.0853	85.3	85.3
	0.1	1 day	0.093, 0.097, 0.091	0.0937	93.7	104
	0.1	3	0.058, 0.059, 0.062	0.0597	59.7	107
	0.1	6	0.082, 0.071, 0.072	0.075	75.0	98.7
	0.1	12	0.098, 0.096, 0.084	0.0927	92.7	129
	0.1	18	0.093, 0.096, 0.089	0.0927	92.7	97.5
Beef Fat–GC/MSD	0.1	1 day	0.104, 0.109, 0.087	0.100	100	100
	0.1	3	0.079, 0.082, 0.081	0.0807	80.7	106
	0.1	6	0.091, 0.076, 0.079	0.0820	82.0	82.0
	0.1	12	0.093, 0.117, 0.128	0.113	113	113
	0.1	18	0.112, 0.118, 0.114	0.115	115	115

*Corrected for mean concurrent recovery (see TABLE C.1.).

D. CONCLUSION

An acceptable analytical method was used to analyze samples of beef kidney, liver, fat, muscle, and milk. Residues of dicamba and DCSA are stable in livestock matrices for 18 months.

E. REFERENCES

MRID No. 46668101; Formanski, L. J. (1996) "Stability of Dicamba and 3,6-Dichlorosalicylic Acid in Stored Frozen Beef Tissues and Milk". Lab Project Number: 480068. Report No. 151. Study No. DP-304489. Unpublished study prepared by Sandoz Agro, Inc. 275 pages

F. DOCUMENT TRACKING

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